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Indian Standard

METHODS OF SAMPLING AND TEST FOR *KATTHA*

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NEW DELHI 110002

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METHODS OF SAMPLING AND TEST FOR *KATTHA*

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Indian Standard

METHODS OF SAMPLING AND TEST FOR *KATTHA*

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 23 October 1964, after the draft finalized by the *KATTHA*, Vegetable Tans and Allied Products Sectional Committee had been approved by the Chemical Division Council.

0.2 *KATTHA* is obtained by crystallization from the extract of the heartwood of *Acacia catechu* Willd fam. Leguminosæ called *KHAIR* in Hindi. The manufacture of *KATTHA* is carried out in India on both small and large scale. *KATTHA* is one of the principal ingredients used in the preparation of *PAN* (betel leaves) for chewing purposes; in combination with lime, it gives the characteristic red colouration on chewing. *KATTHA* is regarded as an astringent; cooling and digestive; useful in relaxed conditions of the throat, mouth and gums; and also in cough and diarrhoea. Externally, it is employed as an astringent and as a cooling application to ulcers, boils and eruptions on the skin. *KATTHA* also enters into a number of *Ayurvedic* and *Unani* medicinal preparations.

0.3 Accepted methods for analysis of *KATTHA* are not available in literature. The Sectional Committee responsible for the preparation of this standard, therefore, referred to whatever varying, or even conflicting, papers had been published on the subject; in that connection, the publication 'A note on the analysis of cutch and the preparation of pure catechin' [*Indian Forester Memoirs*, Vol I, Part 1, (1908)] by Puran Singh was found valuable. The Committee has formulated these methods of test on the basis of the accumulated experience of various Public Analysts in this country as well as actual analyses made by a group of collaborating laboratories, namely, The Public Analyst, Delhi; The Public Analyst, Uttar Pradesh; The Public Analyst, Punjab; Italab Private Ltd., Bombay; The Indian Wood Products Co. Ltd., Bareilly; and The Gwalior Forest Products Ltd., Shivpuri. The co-operation received is gratefully acknowledged.

0.3.1 In this standard, catechin determination (*see* 7) is carried out by extraction with a mixed organic solvent. The Committee considered it desirable to prescribe a melting point test for the purity of the isolated catechin but found that adequate data are not available. The Committee has initiated steps for the collection of this information.

0.4 For particle size, Indian Standard Sieves conforming to IS : 460 - 1962[*] are prescribed. Where IS Sieves are not available, other equivalent standard sieves as judged by aperture size may be used.

0.5 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2 - 1960[†].

1. SCOPE

1.1 This standard lays down the methods of sampling and test for *KATTHA*.

2. SAMPLING

2.1 General Requirements of Sampling

2.1.0 In drawing, preparing, storing and handling samples, the following precautions and directions shall be observed.

2.1.1 Samples shall not be taken in a place exposed to dampness and in insanitary conditions.

2.1.2 Precautions shall be taken to protect the material being sampled, the samples, the sampling instrument and the containers for samples from adventitious contamination, particularly from absorption of water.

2.1.3 The samples shall be placed in suitable, clean, dry and air-tight glass or other suitable containers on which the material has no action.

2.1.4 Each sample container shall be sealed air-tight with a suitable stopper after filling, and marked with full particulars of the material and the date of sampling.

2.1.5 Samples shall be stored in a cool and dry place.

2.2 Scale of Sampling

2.2.1 *Lot* — The material offered for inspection at one time shall constitute a lot.

2.2.1.1 Samples shall be tested for each lot for ascertaining conformity of the material to the requirements of the relevant material specification.

2.2.2 The number (n) of packages to be chosen from a lot shall depend on the number of packages in the lot (N) and shall be in accordance with Table 1.

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[*] Specification for test sieves (*Since revised*).

[†] Rules for rounding off numerical values (*revised*).

TABLE 1 NUMBER OF PACKAGES TO BE SELECTED FOR SAMPLING

(*Clause* 2.2.2)

NO. OF PACKAGES IN THE LOT N (1)	NO. OF PACKAGES TO BE CHOSEN n (2)
3 to 15	3
16 „ 40	4
41 „ 65	5
66 „ 110	7
Over 110	10

2.2.3 These packages shall be chosen at random from the lot. To ensure the randomness of selection, a random number table as agreed to between the purchaser and the supplier shall be used. In case such a table is not available, the following procedure shall be adopted:

> Arrange all the packages in the lot in a systematic manner and starting from any package, count them as 1, 2, 3, etc............up to r and so on, where r is equal to the integral part of the value of N/n. Every rth container thus counted shall be separated until the requisite number of packages is obtained to give the samples for test.

2.3 Preparation of Samples

2.3.1 Draw representative portions of the material from different parts of each package selected for sampling, so as to obtain not less than 200 g of material. In case the proportion of fines in the material so taken out does not exceed 1 percent by weight, the material shall be crushed to powder form and processed further as under **2.3.3**, **2.3.4** and **2.3.5**. For this purpose, fines shall constitute the portion of the material passing through 2·00-mm IS Sieve (*see* IS : 460 - 1962*).

2.3.2 In case the proportion of fines as obtained under **2.3.1** exceeds 1 percent by weight, the entire contents of the package shall be taken out and passed through 2·00-mm IS Sieve. The lumps and fines so separated shall then be weighed and their proportion determined.

2.3.2.1 The lumps after weighing shall be quartered so as to obtain not less than 200 g of material and the latter shall be crushed to pass through 2·00-mm IS Sieve. The fines after weighing shall also be separately quartered so as to obtain not less than 100 g of material.

2.3.2.2 Proportionate weights of lumps and fines as determined under **2.3.2** shall be taken from the portions obtained under **2.3.2.1** and thoroughly mixed together to obtain a total of about 200 g of material.

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*Specification for test sieves (**Since revised**).

2.3.3 *Composite Sample* — Out of the material obtained under **2.3.1** or **2.3.2.2**, as the case may be, a small but equal quantity shall be taken from each selected package and shall be well mixed up together so as to form a composite sample of not less than 150 g for the lot. This composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee.

2.3.4 *Individual Samples* — The remaining portion of the material from each package (after the small quantity needed for the formation of composite sample has been taken out) shall be divided into three equal parts, each part weighing not less than 50 g. These parts shall be transferred immediately to thoroughly dried bottles, closed with air-tight glass stoppers and labelled with all the particulars of sampling. The material in each such closed bottle shall constitute an individual sample. These individual samples shall be separated into three identical sets in such a way that each set has an individual sample representing each of the packages selected. One of these sets shall be marked for the purchaser, another for the supplier and the third for the referee.

2.3.5 *Referee Samples* — Referee samples shall consist of a composite sample and a set of individual samples marked for this purpose and shall bear the seals of the purchaser and the supplier. These shall be kept at a place agreed to between the two and shall be used in case of a dispute.

3. QUALITY OF REAGENTS

3.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070 - 1960*) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

4. PREPARED SAMPLE

4.1 Grind the sample in a porcelain mortar and pestle or other suitable grinder so that it completely passes through 425-micron IS Sieve (*see* IS : 460 - 1962†). This shall be the ***prepared sample*** and shall be used for tests.

5. TEST FOR STARCH

5.0 Outline of the Method — A solution of the material is tested with iodine.

5.1 Reagent

5.1.1 *Iodine Solution* — Dissolve 2·6 g of iodine and 3 g of potassium iodide in a sufficient quantity of water to obtain 100 ml.

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*Specification for water, distilled quality (*revised*). (Since revised).
†Specification for test sieves (Since revised).

5.2 Procedure — Boil about 1 g of the *prepared sample* with 10 ml of water for about 1 minute; allow to cool and add a few drops of iodine solution.

5.2.1 The material shall be considered to be free from starch if no distinct blue colour is developed.

6. LOSS ON DRYING

6.1 Procedure — Weigh accurately about 2 g of the *prepared sample* in a tared weighing bottle or petri dish and dry in an oven at 98° to 100°C for 3 to 4 hours. Repeat heating, cooling and weighing till constant weight (constant to ± 2 mg) is obtained.

6.2 Calculation

$$\text{Loss on drying, percent by weight} = \frac{100(W_1 - W_2)}{W_1}$$

where

W_1 = weight in g of the *prepared sample* taken for the test, and
W_2 = weight in g after drying.

7. CATECHIN

7.0 Outline of the Method — The catechin is extracted out by refluxing with a mixture of carbon disulphide and ethyl ether; the extract is evaporated to dryness and weighed.

7.0.1 The catechin content signifies the efficiency of separation of (crystallized out) *KATTHA* from the mother liquor in the manufacturing process.

7.1 Reagents

7.1.1 *Methyl Alcohol* — conforming to IS : 517-1954*.

7.1.2 *Solvent Mixture* — prepared by mixing carbon disulphide (conforming to IS : 717 - 1955†) with ethyl ether (conforming to IS : 336 - 1964‡) in the ratio 1 : 4.

7.2 Procedure — Weigh accurately about 1 g of the *prepared sample* in a 100-ml conical flask and shake with 20 ml of methyl alcohol to form a slurry. Evaporate the methyl alcohol on a water-bath. Add 100 ml of the solvent mixture and reflux, using a water condenser, on a water-bath for half an hour. Cool the mixture, allow to settle and filter by decantation through a filter paper (Whatman No. 11 or equivalent) into a beaker. Repeat the process of extraction twice, each time using fresh 100 ml of the

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*Specification for methanol (methyl alcohol). (Since revised).
†Specification for carbon disulphide, technical. (Since revised).
‡Specification for ether (*revised*). (Since revised).

solvent mixture. Wash the residue after the last extraction with 20 ml of the solvent mixture and combine the filtrates and washings in the beaker. Evaporate the combined liquid to about 20 ml and then transfer quantitatively, using small volumes of the solvent mixture for washing the beaker, into a weighed dish. Continue evaporation of the solvent at room temperature and then dry the dish with the residue in an oven at 50° ± 2°C to constant weight (constant to ± 2 mg).

NOTE — In case of difficulty in filtering after refluxing due to high volatility of the solvent mixture, suitable arrangements shall be made to keep the temperature of the contents in the funnel low.

7.3 Calculation

$$\text{Catechin, percent by weight (on dry basis)} = \frac{10\,000\, W_1}{W_2 (100 - M)}$$

where

W_1 = weight in g of the residue,

W_2 = weight in g of the *prepared sample* taken for the test, and

M = percent loss on drying as determined in 6.

8. COLD WATER EXTRACTIVES

8.0 Outline of the Method — The sample is kept for about 72 hours with water at about 3°C. The filtrate is evaporated and the residue weighed.

8.0.1 Cold water extractives indicate the amount of cutch present in *KATTHA*.

8.1 Reagent

8.1.1 *Toluene*

8.2 Procedure — Weigh accurately about 1 g of the *prepared sample* and mix thoroughly in a conical flask with 100 ml of water at about 3°C. Add 1 ml of toluene as a preservative to the flask and keep in the refrigerator at about 3°C for 72 hours. Shake the flask occasionally during this period. Filter the supernatant liquid through a filter paper (Whatman No. 11 or equivalent) under vacuum. Stir the residue with a further quantity of water at about 3°C and filter until no more colour is extracted from the residue. Combine the filtrate and the washings and transfer to a tared porcelain dish. Evaporate the water at 95° ± 2°C. Dry the residue at 98° to 100°C to constant weight (constant to ± 2 mg).

8.3 Calculation

$$\text{Cold water extractives, percent by weight (on dry basis)} = \frac{10\,000\, W_1}{W_2 (100 - M)}$$

where

W_1 = weight in g of the residue,

W_2 = weight in g of the *prepared sample* taken for the test, and

M = percent loss on drying as determined in 6.

9. MATTER INSOLUBLE IN RECTIFIED SPIRIT

9.0 Outline of the Method — The sample is treated with rectified spirit at 37°C for 24 hours, the residue is dried and weighed.

9.1 Reagent

9.1.1 *Rectified Spirit* — conforming to IS : 323 - 1959*.

9.2 Procedure — Weigh accurately about 1 g of the *prepared sample* and macerate with 100 ml of rectified spirit. Shake frequently for 6 hours and then allow to stand for 18 hours at 37° ± 2°C. Filter the supernatant liquid on a filter paper (Whatman No. 11 or equivalent) and wash the residue free of soluble matter with rectified spirit maintained at 37° ± 2°C. Washing of the residue shall be continued till the filtrate leaves no residue on evaporation. Dry the residue with the filter paper in a tared weighing bottle at 98° to 100°C for 3 to 4 hours, cool in a desiccator and weigh, using a filter paper similarly treated as counterpoise. Repeat heating, cooling and weighing until constant weight (constant to ± 2 mg) is obtained.

NOTE — Filtration may be done with a sintered glass crucible (G No. 4) or a Gooch crucible with asbestos bed as an alternative.

9.3 Calculation

Matter insoluble in rectified spirit, percent by weight (*on dry basis*) $= \frac{10\,000\,W_1}{W_2(100-M)}$

where

W_1 = weight in g of the residue,

W_2 = weight in g of the *prepared sample* taken for the test, and

M = percent loss on drying as determined in 6.

10. WATER INSOLUBLE SOLIDS AT 37° ± 2°C

10.1 Procedure — Weigh accurately about 1 g of the *prepared sample* in a conical flask and add 100 ml of water maintained at 37° ± 2°C to it. Shake the flask frequently for about 6 hours and then allow it to stand for 18 hours at 37° ± 2°C. Filter the supernatant solution through a filter paper (Whatman No. 11 or equivalent) under vacuum and wash the residue with water (at 37° ± 2°C) until washings are colourless. Dry the residue with the filter paper at 98° to 100°C in a weighing bottle to constant weight (constant to ± 2 mg), using a filter paper similarly treated as counterpoise.

NOTE — Filtration may be done with a sintered glass crucible (G No. 4) or a Gooch crucible with asbestos bed as an alternative.

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*Specification for rectified spirit (*revised*).

10.2 Calculation

$$\text{Water insoluble solids at } 37° \pm 2°\text{C, percent by weight (on dry basis)} = \frac{10\,000\,W_1}{W_2\,(100 - M)}$$

where

W_1 = weight in g of the residue,

W_2 = weight in g of the *prepared sample* taken for the test, and

M = percent loss on drying as determined in **6**.

11. BOILING WATER INSOLUBLE SOLIDS

11.1 Procedure — Weigh accurately about 1 g of the *prepared sample* and reflux with 100 ml of water for one hour. Filter the supernatant liquid through a filter paper (Whatman No. 11 or equivalent) under vacuum, using a hot water jacket funnel. Wash the residue with boiling water until washings are colourless. Dry the residue with the filter paper in a weighing bottle at 98° to 100°C to constant weight (constant to ± 2 mg), using a filter paper similarly treated as counterpoise.

NOTE — Filtration may be done with a sintered glass crucible (G No. 4) or a Gooch crucible with asbestos bed as an alternative.

11.2 Calculation

$$\text{Boiling water insoluble solids, percent by weight (on dry basis)} = \frac{10\,000\,W_1}{W_2\,(100 - M)}$$

where

W_1 = weight in g of the residue,

W_2 = weight in g of the *prepared sample* taken for the test, and

M = percent loss on drying as determined in **6**.

12. TOTAL ASH

12.1 Procedure — Weigh accurately about 2 g of the *prepared sample* in a tared platinum or silica dish and incinerate at dull red heat in a muffle furnace or over a Bunsen flame till no carbonaceous particles are visible. Allow it to cool and weigh. Repeat heating, cooling and weighing till constant weight (constant to ± 2 mg) is obtained. Preserve the ash for test in **13**.

12.2 Calculation

$$\text{Total ash, percent by weight (on dry basis)} = \frac{10\,000\,W_1}{W_2\,(100 - M)}$$

where

W_1 = weight in g of the ash obtained,

W_2 = weight in g of the *prepared sample* taken for the test, and

M = percent loss on drying as determined in **6**.

13. ASH INSOLUBLE IN HYDROCHLORIC ACID

13.1 Reagent

13.1.1 *Dilute Hydrochloric Acid* — 1 : 2·5 (*v/v*).

13.2 **Procedure** — To the dish containing the preserved ash (**12.1**), add 25 ml of dilute hydrochloric acid. Cover the dish with a watch-glass and boil the contents carefully for 5 minutes. Cool the solution and filter through a filter paper (Whatman No. 11 or equivalent). Wash the residue with hot water until the filtrate is neutral to litmus. Transfer the filter paper along with the residue to the dish and incinerate completely. Cool the basin and weigh. Repeat heating, cooling and weighing till constant weight (constant to ± 2 mg) is obtained.

13.3 Calculation

$$\text{Ash insoluble in hydrochloric acid, percent by weight (on dry basis)} = \frac{10\,000\, W_1}{W_2\,(100 - M)}$$

where

W_1 = weight in g of the ash insoluble in hydrochloric acid,

W_2 = weight in g of the *prepared sample* taken for the test in **12.1**, and

M = percent loss on drying as determined in **6**.

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